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(21) International Application Number: PCT/HU89/00041 (22) International Filing Date: 11 August 1989 (11.08.89) (30) Priority data: 4310/88 11 August 1988 (11.08.88) HU 4310/88 27 April 1989 (27.04.89) HU (71)(72) Applicants and Inventors: TÓTHNÉ PÁPAY, Valéria [HU/HU]; TÓTH, Valéria, Katalin [HU/HU]; TÓTH, Tünde [HU/HU]; Petőfi S. sugárút 40. D/II/3, H-6722 Szeged (HU). (74) Agent: DANUBIA; P.O. Box 198, H-1368 Budapest (HU). (81) Designated States: AT (European patent), BE (European patent), CH (European patent), DE (European patent), FR (European patent), GB (European patent), IT (Euro- pean patent), JP, LU (European patent), NL (European patent), SE (European patent), US.		Published <i>With international search report.</i>
(54) Title: PROCESS FOR PRODUCING LIGHT- AND HEAT-RESISTANT, BIOLOGICALLY ACTIVE EXTRACTS, JUICES AND PRODUCTS RICH IN VITAMIN C, FLAVOUR, AROMA AND COLOURING SUBSTANCES, AS WELL AS FOR PRODUCING PHARMACEUTICALS, COSMETICS, FOODSTUFF, NUTRIMENTS, FODDER AND MATERIALS YIELDING BIOLOGICAL PROTECTION CONTAINING THE ABOVE EXTRACTS, JUICES AND PRODUCTS AS ACTIVE INGREDIENTS		
(57) Abstract The invention relates to the production of biologically active extracts rich in vitamin C and aroma substances and resistant to light and heat. According to the process of the invention one or more plant or plant-parts containing 0.1 to 99 % by weight of natural colouring substances, acids and/or antioxidants are extracted together or separately cold and/or warm for 1 minute to several days, at a temperature of -20°C to +200°C, optionally at an elevated or reduced pressure, optionally in a CO ₂ , nitrogen oxide or acetic acid atmosphere, with or without flavour or aroma substances, salts, acids, bases, sugars, water or organic solvents, and the treatment optionally is once or more times repeated. The process according to the invention enables the preparation of biologically active, light- and heat-resistant extracts rich in vitamin C, aroma substances and active ingredients from easily available plants. The extracts obtained by the process according to the invention can be used for medicines, cosmetics, foodstuffs, further as nutritives, additives to fodder, for promoting germination or as sprays for controlling insects.		

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PROCESS FOR PRODUCING LIGHT- AND HEAT-RESISTANT,
BIOLOGICALLY ACTIVE EXTRACTS, JUICES AND PRODUCTS
RICH IN VITAMIN C, FLAVOUR, AROMA AND COLOURING
SUBSTANCES, AS WELL AS FOR PRODUCING PHARMACEUTICALS,
5 COSMETICS, FOODSTUFF, NUTRIMENTS, FOODER AND MATERIALS
YIELDING BIOLOGICAL PROTECTION CONTAINING THE ABOVE
EXTRACTS, JUICES AND PRODUCTS AS ACTIVE INGREDIENTS

The invention relates to a process for producing
10 light- and heat resistant biologically active extracts,
juices and products rich in vitamin C, flavour, aroma
and colouring substances, as well as for producing
pharmaceuticals, cosmetics, foodstuff, nutriments,
fodder and materials yielding biological protection
15 containing the above extracts, juices and products as
active ingredients.

In the process of the invention the extraction
methods are chosen taking into consideration the
biological and chemical characteristics of the plants,
20 the factors influencing the pH-value and the properties
of the plants containing colouring substances; the
extraction method is combined with the utilization of
the pH influencing property of other plants and with
physical methods. In this way light- and heat-resistant
25 coloured extracts and juices may be obtained.

By using known methods of preservation the
process according to the invention assures the preserva-
tion of the active ingredients of plants with seasonal
character and their continuous supply without reducing
30 the amount of the active ingredients, flavour and aroma.

In course of processing and storage the colour,
aroma and vitamins may change, become decomposed and
transformed, so they do not meet the requirements in
respect to quality and aesthetics, they are unsuitable
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for consumption, simultaneously losing their value.

For colouring foodstuffs and pharmaceuticals only natural substances can be used and it is necessary that the colour obtained be permanent and the colouring materials should not interfere with the active ingredients.

Using the known processes it has repeatedly been observed that in course of washing, soaking and blanching vitamin C and a considerable part of the aroma substances get lost or decomposed the loss takes out already at room temperature 8 to 50%. (Kubat K., Vajda P., Hajdu I., Lindner K.: Konzerv- és Paprikaipar (Conserve and paprika industry), 5, 121-124 (1976); Jin A., Li T., Zhi G.: Skipin Kexue (Beijing) 43, 20, (1983).

In HU-PS 182,314 the adjustment of the pH of vegetables to 4.3 - 4.5 is proposed for preserving their colour and vitamin content.

According to the Hungarian Patent HU-PS 186,152 juices of apple and rose-hips are produced using cell-explosion accompanied by gas diffusion under a high pressure and in the presence of carbon dioxide and nitrogen oxide. The fruits used are premoistened and presoaked.

In HU-PS 186,495 for producing fruit juices extracting agents of pH 3.26 are used together with enzymes promoting the decomposition.

The solution according to the Hungarian Patent HU-PS 192,817 uses oil containing vitamin E, vitamin C and phosphatides preserving by this the colour of paprika.

In HU-PS 185,706 it is described that the colour of paprika is preserved by using carbon dioxide and ethane at a high pressure and at a temperature of 40 to 70 °C.

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The known processes could not eliminate the loss of vitamin C occurring in course of washing, soaking, blanching, freezing or recovery of juice, they are unable to reduce the browning resulting from enzyme function or decolourization. The decolourization occurring in course of preservation is eliminated by sterilization.

The invention is based on the recognition that the decomposition of vitamin C is followed in the whole course of the processing procedure up to the obtaining of the final product, at the same time methods assuring maximal recovery of vitamin C are inserted into the extraction procedure and simultaneously natural active ingredients and biological methods are used to diminish the oxidation and decomposition.

The invention is based on the recognition that the efficiency of the recovery of active the ingredients can be increased if a cold or warm extraction is performed in more steps in such a way that at first the extraction is continued for 5 to 20 min. up to 48 hours, preferably for 15 min. to 2.5 hours (in case of fermentation for 8 to 14 days), thereafter warm extraction is performed repeatedly for 10 minutes to 4 hours at the boiling point of the solvent or in a water bath, i.e. from -4°C to 100°C ; then the product is immediately filtered and compressed, the compressed drug is repeatedly extracted at a pressure of 40 to 180 KPa or at an atmospheric pressure. The common duration of the two warm extractions should amount to at least four hours; then the extracts are fractionated with solvents in the presence of electrolytes.

Furtheron, the invention is based on the recognition that the efficiency of the extraction can be increased by neutralization of the components promoting

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the oxidation and contained in the extractive agent or in the auxiliary materials or by preventing the oxidation.

For this purpose products of apiculture, materials with anti-oxidizing effect, acids or salts can be used.

Further, the invention is based on the recognition that the efficiency of the juices and extracts obtained by the process according to the invention can be further increased, to the extracts additional therapeutic effects can be given by the simultaneous use of known medical herbs, active ingredients and products of apiculture.

Summing up, the invention relates to a process for producing biologically active light- and heat-resistant extracts, juices and products rich in vitamin C, flavour, aroma and colouring substances, as well as for producing pharmaceuticals, cosmetics, foodstuff, nutriments, fodder and materials yielding biological protection containing the above extracts, juices and products as active ingredients.

In accordance with the invention fresh, frozen or dried plants or plant-parts containing natural colouring substances, acids and/or antioxidants (flavonoide, anthocyan, carotenoid, vitamin E) in an amount of 0.1 to 99.9 % by weight and active ingredients together or separately are extracted at an elevated, reduced or atmospheric pressure, in CO₂, nitrogen oxide or ethane atmosphere or in water vapour, acetic acid, formic acid or mixed formic acid - hydrogen peroxide atmosphere cold and/or warm for 1 minute to several days at a temperature of -70 °C to +200 °C, preferably at -4 °C to 120 °C with one or more organic solvent and/or

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- water, glycerine, 1,2-propylene glycol or with their mixture and/or with aqueous solutions of salts, acids or bases, the extraction is carried out ones or more times after each other, optionally in the presence of
- 5 flavour or aroma substances, sweetenings, antioxidants, enzymes and preservatives at a pH of 2 to 11, then optionally are sterilized at a temperature of 85 °C to 120 °C for 20 to 50 minutes, preferably for 25 minutes, then optionally are adsorbed onto a material of carbo-
- 10 hydrate basis or onto alkaline earth metal salts then they are dried and the extract is formulated in se or together with active ingredients in a known way to compositions and in case of separately performed extrac-tion the extracts are admixed in adequate proportion.
- 15 The extraction may be performed by soaking, mixing, shaking in a shaker or counter-current solvent extraction, steaming or cooking in a Papin-vessel or using microwaves. The extract can also be prepared by fermenta-tion using sugar, salt or yeast, the extracts are prepared in a
- 20 fermentation process of 8 to 14 days.
- As acids citric acid, tartaric acid, malic acid, formic acid, lactic acid, succinic acid, fumaric acid, glucuronic acid, sulphurous acid, phosphoric acid, acetic acid and hydrochloric acid can be used.
- 25 As salts sodium carbonate, calcium hydroxide, sodium hydroxide, sodium metabisulfite, potassium meta-bisulfite, sodium thiosulfate or sodium chloride can be used.
- As preservatives antioxidants potassium sorbate,
- 30 sorbic acid, benzoic acid, sodium benzoate, propylene glycol, polysorbate 80, propyl gallate, vitamin E, fatty acids, L-epicatechine, as well as propolis, extracts

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of poplar buds and of berries of Hippophae rhamnoides L. and extracts containing vitamin E can be used.

As organic solvents those usually applied in therapeutics can be used, certain active constituents
5 can be separated by solvent fractionation, in this way the efficiency can be increased.

The efficiency can also be increased by using herbs containing volatile oils, products of apiculture, propolis, royal jelly, honey, pollen and honeycomb.

10 The extracts can be used with or without sterilization directly or they can be concentrated into jams, jellies or pulverized products.

The main advantages of the process according to the invention are as follows:

15 a) it can be carried out easily, the process is simple, economical, energy sparing.

b) In comparison to the known processes extracts or canned vegetables or fruits rich in vitamin C, flavour, aroma and colouring substances can be produced
20 with increased efficiency, and these products can be stored without any change or decomposition or can be transformed into the product directly.

c) Fresh, frozen and dried vegetable-parts can be processed together or separately in whole pieces or
25 previously cut or crushed.

d) Using different simple operations extracts, concentrates or pulverized products of increased efficiency and amount may be produced; these serve as starting materials for basic materials, active ingredients, so
30 e.g. pectins, proteins, polysaccharides, sugar alcohols and concentrates of colouring substances.

The invention will be detailed by means of examples

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as follows:

Example 1

Production of extracts and juices rich in vitamin C,
flavour and aroma materials

1000 g fruit, vegetable part to be processed,
consisting of 200 g of raspberries 200 g of gooseberries
200 g of currants, 200 g of sour-cherries and 200 g of
apples are washed as a whole, peeled if necessary, stoned
and crushed. Thereafter the mass obtained is mixed with
5 g of citric acid, 4.2 g of lactic acid, 0.5 g of sodium
pyrosulfite, 100 g of propylene glycol, 10 g of sodium
bicarbonate and 300 g of sugar and agitated with 1000 g
of water for 25 minutes then pressed, then is mixed with
1000 g of water and is heated in microwaves up to boiling,
then filtrated an pressed. The procedure is repeated
using 1000 g water. The combined extracts are adjusted
to pH 6 to 6.5 with sodium bicarbonate or with hydro-
chloric acid, then sterilized at 85 °C to 98 °C for 30
minutes.

The juice obtained can be used as refreshment, as
extract or concentrate in form of syrup, jam or dried
to a pulverized form, adsorbed onto cyclodextrine, sugar,
salt or alkaline-earth metals.

To obtain colouring substances the protein is
mixed with alcohol in a tenfold quantity, the polysaccharides
are allowed to precipitate, the alcohol is destillated
from the alcoholic extract and after having acidified
the solution, the colouring substances are shaken out
with n-butanol.

The butanolic extract containing colouring
substance is concentrated and used for colouring and
flavouring cosmetics, medicines, foodstuff and confectionery.

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- The residue of the pressing is mixed with 5 % by weight of sulphuric acid and in a twentyfold quantity cation-free water is added; thereafter it is boiled at a temperature of 80 °C for 15 minutes, pressed and the solution is allowed to pass through Varion KS ion-exchanging resin and a discolouring column; then the pectin is precipitated from it with 20 % by weight of aluminium sulfate and 10 % by weight of sodium hydrocarbonate at pH 4.2; then is dehydrated with alcohol, the pectin is released with hydrochloric acid, cleaned by centrifuging, concentrated, dried to 60-65 % by weight of dry substance content, admixed with 25 % by weight of citric acid, dried to 80-90 % by weight of extract content and pulverized.
- 15 Skin and core of the apples are used for producing pectin. Stones of raspberry, goosberry and sour-cherry remaining back after pressing are ground and heated to boiling with a mixture of concentrated formic acid and hydrogen peroxide in a ratio of 8 to 1 using
- 20 microwaves so the oil gets separated from the stones. The material obtained is applied admixed to fodder to make use of its high protein and fat content.

Example 2

- 25 Producing frozen vegetable and fruit with high vitamin content

- Vegetables and fruits to be processed are washed in water saturated with carbon dioxide, cut to pieces, thereafter the vegetable part to be frozen is treated
- 30 in carbon dioxide vapour for 3 to 8 minutes, then preserved by mixing with 4 o/oo of citric acid, 5 o/oo of sodium pyrosulfite and 10 % of sugar or 2.5 % of salt, then is frozen or drie-frozen or sterilized at a

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temperature of 98 °C under overpressure and using pouring on liquid.

Example 3

5 Producing an extract improving the mental activity and the sight

200 g of raspberries, 200 g of cranberries, 200 g of pritamin paprikas, 200 g of carrots, 200 g of black currants, 200 g of red beets, 200 g of rose-hips, 50 g of petals of peony, 50 g of petals of red rose are washed in water containing carbonic acid, are crushed and comminuted, thereafter are shaken in a shaker with 150 g pollen, 3000 g sodium pyrosulfite solution of 4 o/oo, 50 g tartaric acid, 200 g glucose and 200 g fructose
15 for 15 minutes, thereafter it is pressed, filtered and the residue is boiled with 1500 g water using microwaves; then it is pressed, the product is cooked with further 1500 g water in a Papin-vessel for four hours and thereafter is pressed. The liquid pressed out is preserved
20 with 1 % propolis and 1 o/oo K-sorbate and is sterilized at 95 °C for 25 minutes. The final product can be used as a syrup, drink, jam or in a pulverized form for medicines, cosmetics, confectioneries, as a refreshment, as additive to fodder for increasing the germination
25 and in products of repellent effect for repelling insects. The product improves the sight, improves the mental activity, activates, regenerates the complexion, cures wounds, replaces vitamins and is suitable for producing colouring substances and aromae.

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Example 4

Producing an extract for excrement regulation

- 500 g of rhubarbs, 500 g of rhubarb stalks,
250 g of baked pumpkins, 250 g of black currants,
5 250 g of medlars, 500 g of apples are mixed with 50 g
of calcium carbonate and 2500 g of water. Thereafter
the mass is heated to boiling is filtered and pressed;
then 2500 g of Malaga wine, 1000 g of sugar, 250 g
of lemon juice, 100 g of lemon peel, 100 g of orange peel,
10 20 g of cinnamon, 20 g of vanilla, 20 g of cardamom
seeds are added, the mixture is heated up to boiling and
kept on slow fire for 20 minutes, then it is filtered
and pressed. The drink obtained has slight laxative
effect, stimulates the digestion, is rich in minerals,
15 reduces inflammations and has an antispasmodic effect.
To promote the laxative effect flax-seed can be added.

Example 5

Meta-drink

- 20 250 g of rose-hips, 250 g of raisins, 150 g of
malt grits, 100 g of citric acid, 50 g of rosemaries
50 g of hibiscus flowers, 200 g of beer-yeast and 15 kg
of honey are cooked with 40 l of water up to foaming,
and the mixture is fermented at a temperature of 25 °C
25 for 4 weeks, thereafter it is flavoured with 50 g of
cloves, 50 g of allspices, 50 g of nutmegs, 50 g of nut-
megflowers, 20 g of gingers, 30 g of cinnamons and 30 g
vanilla; after completing the fermentation the mucilage
and the protein is removed from the beer by mixing the
30 beer with calcium carbonate and egg-white.

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We claim:

1. Process for producing biologically active light- and heat-resistant extracts, juices and products rich in vitamin C, flavour, aroma and colouring substances, as well as for producing pharmaceuticals, cosmetics, foodstuff, nutriment, nutriment, fodder and materials yielding biological protection containing the above extracts, juices and products as active ingredients by extracting fresh, frozen or dried plants or plant-parts containing natural colouring substances (flavonoids, anthocyan, carotenoid), acids and/or antioxidants in an amount of 0.1 to 99.9 % by weight and active ingredients together or separately are extracted at an elevated, reduced or atmospheric pressure, in CO₂, nitrogen oxide or ethane atmosphere or in water vapour, acetic acid, formic acid or mixed formic acid - hydrogen peroxide atmosphere cold and/or warm for 1 minute to several days at a temperature of -70 °C to +200 °C, preferably at +4 °C to 120 °C with one or more organic solvent and/or water, glycerine, 1,2-propylene glycol or with their mixture and/or with aqueous solutions of salts, acids or bases, the extraction is carried out one or more times after each other, optionally in the presence of flavour or aroma substances, sweeteners, antioxidants, enzymes and preservatives at a pH of 2 to 11, then optionally are sterilized at a temperature of 85 °C to 120 °C for 20 to 50 minutes, preferably for 25 minutes, then optionally are adsorbed onto a material of carbohydrate basis or onto alkaline-earth metal salts then they are dried and the mixture is formulated in or together with active ingredients in a known way to

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compositions and in case of separately performed extraction the extracts are admixed in adequate proportion.

2. Process for producing preparations for human and veterinary therapeutics, characterized in that the extract(s) produced according to claim 1 are mixed with each other and/or with carriers, adjuvants, usually applied for the preparation of medicines and are formulated into different forms of medicines (solutions, powders, syrups, suppositories, ointments, dusting-powders, injections, tablets etc.).

3. Process for producing cosmetics, characterized in that the extracts produced according to claim 1 are mixed with one another and/or with carriers, adjuvants usually applied for the preparation of cosmetics and are formulated into different forms of cosmetics.

4. Process as claimed in claim 1, characterized in that the extract obtained may be used with or without ballast material for fodders.

5. Process as claimed in claim 1, characterized in that mixed or unmixed concentrates of foodstuffs are added in compliance with the target set, and in such a manner finished or semi-finished foodstuffs are prepared.

6. Process as claimed in claim 1, characterized in that the extracts are mixed with one another and/or with carriers usually applied for producing confectionary products.

7. Process as claimed in claim 1, characterized in that the product is used as a biologically protective solution or as a nutritive solution.

8. Process as claimed in claim 1, characterized in that all parts of the raw material to be processed are utilized separately or together

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with the by-products for fodder, as heating material,
for recovery of basic materials or for producing
compost.


9. Process as claimed in claim 1,
characterized in that the extract serves as
basic material for obtaining active ingredients and
dyestuffs.

10. Process as claimed in claim 1,
characterized in that vegetable and fruit cans
are processed in the canned food industry or deep-frozen
products are prepared in the cold-storage industry.

11. Process as claimed in claim 1,
characterized in that starting materials for
active ingredients, aromae, as well as for products
for refreshments, beer, liqueur, aromae and other
products of the distilling industry are produced.

INTERNATIONAL SEARCH REPORT

International Application No PCT/HU 89/00041

I. CLASSIFICATION OF SUBJECT MATTER (If several classification symbols apply, indicate all) * According to International Patent Classification (IPC) or to both National Classification and IPC IPC 5: A 23 L 1/00, 2/00; A 61 K 7/00, 35/78; A 23 K 1/14; A 23 G 3/00		
II. FIELDS SEARCHED Minimum Documentation Searched * Classification System Classification Symbols Int.Cl. 5 A 23 L 1/00, 1/212, 1/221, 1/03, 2/00; A 61 K 7/00, 35/00, 35/78; A 23 K 1/00, 1/14; A 23 G 3/00 Documentation Searched other than Minimum Documentation to the Extent that such Documents are Included in the Fields Searched * Relevant basic textbooks and monographs; WPI/L		
III. DOCUMENTS CONSIDERED TO BE RELEVANT *		
Category *	Citation of Document, ** with indication, where appropriate, of the relevant passages **	Relevant to Claim No. **
X	J. Schormüller "Handbuch der Lebensmittel-chemie", Volume 1, published 1965, by Springer Verlag (Berlin, Heidelberg, New York), see pages 364-367, 1160-1168.	(1, 2, 4-6, 10-11)
X	H. Trillich "Rösten und Röstwaren", published 1934, by Verlag B. Heller (München), see pages 218, 219.	(1)
X	H. Janistyn "Taschenbuch der modernen Parfümerie und Kosmetik", published 1966, by Wissenschaftliche Verlagsgesellschaft m.b.H. (Stuttgart), see pages 48-51, 76, 77.	(1, 3)
X	W. Foerst "Ullmanns Encyclopädie der technischen Chemie", Volume 6, edition 3, published 1955, by Urban und Schwarzenberg (München, Berlin), see pages 36-42.	(1, 2)
X	"Hagers Handbuch der Pharmazeutischen Praxis" Volume 7/A, edition 4, published 1971, by Springer Verlag (Berlin, Heidelberg, New York), see pages 25-33, 308.	(1)
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IV. CERTIFICATION		
Date of the Actual Completion of the International Search 03 November 1989 (03.11.89) International Searching Authority AUSTRIAN PATENT OFFICE		Date of Mailing of this International Search Report 08 November 1989 (08.11.89) Signature of Authorized Officer 

III. DOCUMENTS CONSIDERED TO BE RELEVANT (CONTINUED FROM THE SECOND SHEET)

Category *	Citation of Document, with indication, where appropriate, of the relevant passages	Relevant to Claim No
X	HU, B, 182 314 (NOGRAD MEGYEI ZOELDERT) 28 December 1983 (28.12.83), see totality.	(1)
X	DE, C, 3 114 593 (KRUPP) 09 December 1982 (09.12.82), see claims 1-5; examples.	(1)
